Copyright © 2001 by Humana Press Inc.
All rights of any nature whatsoever reserved.
0273-2289/01/91–93/0253/$13.75

Continuous Countercurrent Extraction of Hemicellulose from Pretreated Wood Residues

Kyoung Heon Kim, Melvin P. Tucker, Fred A. Keller, Andy Aden, and Quang A. Nguyen*

National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden, CO, 80401-3393, E-mail: quang_nguyen@nrel.gov

Abstract

Two-stage dilute acid pretreatment followed by enzymatic cellulose hydrolysis is an effective method for obtaining high sugar yields from wood residues such as softwood forest thinnings. In the first-stage hydrolysis step, most of the hemicellulose is solubilized using relatively mild conditions. The soluble hemicellulosic sugars are recovered from the hydrolysate slurry by washing with water. The washed solids are then subjected to more severe hydrolysis conditions to hydrolyze approx 50% of the cellulose to glucose. The remaining cellulose can further be hydrolyzed with cellulase enzyme. Our process simulation indicates that the amount of water used in the hemicellulose recovery step has a significant impact on the cost of ethanol production. It is important to keep water usage as low as possible while maintaining relatively high recovery of soluble sugars. To achieve this objective, a prototype pilot-scale continuous countercurrent screw extractor was evaluated for the recovery of hemicellulose from pretreated forest thinnings. Using the 274-cm (9-ft) long extractor, solubles recoveries of 98, 91, and 77% were obtained with liquid-to-insoluble solids (L/IS) ratios of 5.6, 3.4, and 2.1, respectively. An empirical equation was developed to predict the performance of the screw extractor. This equation predicts that soluble sugar recovery above 95% can be obtained with an L/IS ratio as low as 3.0.

Index Entries: Extraction; hemicellulose; softwood; pretreatment; acid hydrolysis.

Introduction

In a previous study *(1)*, we concluded that two-stage dilute sulfuric acid pretreatment of softwood forest thinnings gave higher sugar yields than single-stage pretreatment. Figure 1 shows a simplified block-flow

*Author to whom all correspondence and reprint requests should be addressed.

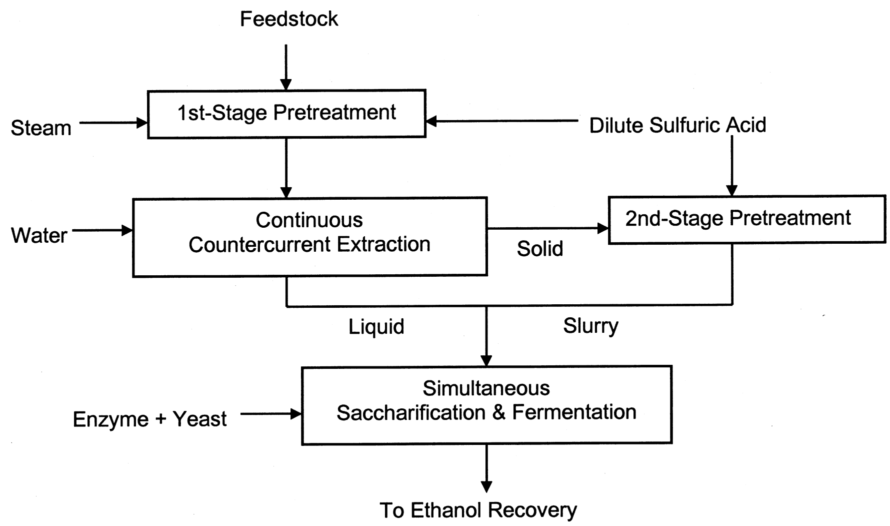

Fig. 1. Two-stage dilute sulfuric acid pretreatment of softwood forest thinnings.

diagram of a two-stage pretreatment process. In this process, high recovery of soluble sugars in the first-stage pretreated material (or hydrolysate) is essential because sugars remaining in the first-stage hydrolysate would be destroyed in the second-stage hydrolysis. Furthermore, the amount of wash water required to achieve high sugar recovery has a significant impact on the process economics. Too much wash water would dilute the sugar stream and increase the cost of fermentation and ethanol distillation. In general, countercurrent washing of the pretreated biomass is required to achieve adequate sugar recovery and high sugar concentration in the extract while maintaining a reasonable water usage requirement. One common term used to describe the amount of water used for extracting solubles from material is the liquid-to-solids (L/S) ratio, which is the ratio of water used in the extraction process over the dry wt of total solids (insoluble and soluble) in the feed. Because the content of soluble solids varies with pretreated materials and diminishes as the solids are being washed, the liquid-to-insoluble solids (L/IS) ratio is also used. The L/IS ratio is essentially constant throughout a countercurrent extractor at steady state, whereas the L/S ratio increases as the soluble solids are removed. We used the L/IS ratio to ensure consistent comparison of extraction characteristics for different pretreated biomass materials.

Figure 2 shows the effect of the L/IS ratio on soluble sugar recovery from first-stage hydrolysate and cost of ethanol production for a 2000 dry t/d softwood-to-ethanol plant using two-stage dilute acid hydrolysis *(2)*. The sugar recovery values were based on the results of three-stage, stagewise, countercurrent extraction experiments *(3)*. The process simulation in Fig. 2 implies that reducing the amount of water used in extracting sugars from first-stage hydrolysate from an L/IS ratio of 5.0 to 3.0 would lower the cost of ethanol production from \$0.34/L (\$1.29/gal) to \$0.31/L

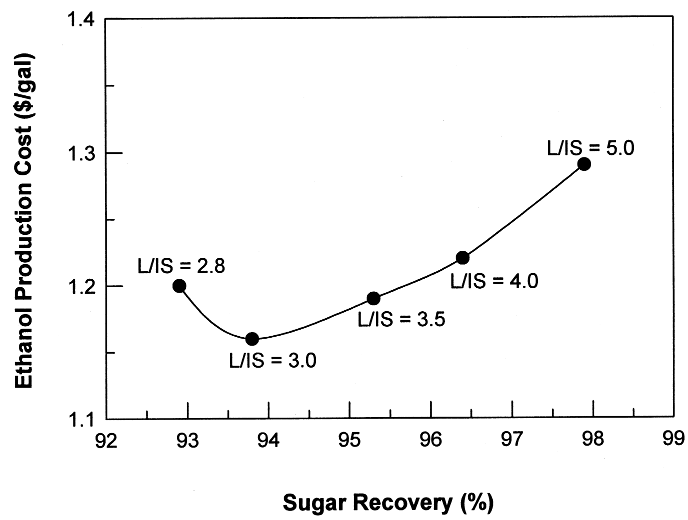

Fig. 2. Effect of L/IS ratio on soluble sugar recovery and production cost of ethanol (2000 dry t/d, $25/dry t softwood residues, two-stage dilute acid hydrolysis process, without enzyme addition).

($1.16/gal), even though the sugar recovery would be lowered from 97.9 to 93.8%. This reduction in production cost is owing mainly to the lower energy requirements to recover ethanol resulting from fermentation of a more concentrated sugar stream. Further reduction of L/IS to 2.8 raises the cost of ethanol production because the negative impact of sugar loss is greater than the savings from lower water usage. If a greater number of extraction stages or a continuous countercurrent extraction device is used, the L/IS ratio can probably be reduced to <3.0 while achieving high sugar recovery and further reducing the cost of ethanol production.

Countercurrent extraction of solubles from biomass materials (such as pulp, sugarcane, fruits, seeds, and pretreated lignocellulose) can be accomplished in a variety of commercial equipment *(4,5)*. The main criteria for selecting countercurrent washing of pretreated biomass to recover soluble sugars include high sugar recovery, high sugar concentration in the extract (i.e., low L/IS ratio), and low capital and operating costs. These criteria are generally the same as those used in the food-processing industry for extraction of sugars and other soluble solids from a variety of feedstock. By comparison, most stagewise washers used in the pulp industry are designed primarily for thorough washing of fibers and not necessarily for obtaining a high concentration of solutes in the wash water. Therefore, our focus is on continuous countercurrent extraction equipment used in the food-processing industry because these systems are most effective in reducing water requirements. Screw conveyors (single or twin) and screw towers are commonly used for extraction of sugar from sugarcane, sugar beet, and fruits *(4,6)*. Pilot-scale screw extractors were used to extract soluble components from sweet sorghum silage *(7)* and steam-pretreated lignocellulosic materials *(8)*. Twin-screw extractors provide better liquid/solid contact and, thus, are generally more efficient than single-screw extractors. However, twin-screw extractors are generally more expensive. The design of intermittent-reversing of screw rotation direction was reported to overcome the compaction problem and inefficient liquid/solid contact in single-screw extractors *(9,10)*. We installed mixing paddles on the auger of our single-screw extractor to improve liquid/solid contact.

Good liquid/solid contact in screw extractors also depends on the drainage characteristics of the pretreated biomass. The particle size of biomass may be important in continuous countercurrent extraction because very fine particles tend to compact and cause liquid to channel or block liquid flow completely. Water temperature may also have an effect on the extraction of solubles from the pretreated biomass. The impact of these parameters and the L/IS ratio on soluble sugar recovery from pretreated softwood forest thinnings was explored in a series of experiments using small column percolators and a pilot-scale single-screw extractor.

Materials and Methods

Pretreated Biomass

Whole-tree chips (passing through a 0.5-in. screen) from California softwood forest thinnings were soaked in 0.66% (w/w) sulfuric acid solution. The acid-impregnated chips were air-dried to 43% (w/w) solids (the acid concentration of liquid in air-dried chips was 1.08% w/w), then pretreated at 185°C for 4 min using a 4-L steam explosion reactor described previously *(1)*. At these conditions, approx 85% of the hemicellulose was solubilized. The water-insoluble fraction of the pretreated material was 72.9% on a dry wt basis. Table 1 gives the feedstock composition and the theoretical component yields after pretreatment. As seen in Table 1, the conversion yield of cellulose (i.e., glucan) was lower than that of hemicellulose (i.e., mannan, galactan, xylan, and arabinan) owing to the mild pretreatment condition aimed at maximizing hemicellulose hydrolysis. The total soluble solids concentration of the liquid fraction of the pretreated material was 99.8 g/L, and the sugar composition of the liquid fraction is given in Table 2.

Yellow poplar sawdust was pretreated at 0.3% (w/w) sulfuric acid and 195°C for 5 min using a Sunds™ Hydrolyzer installed at the Process Development Unit of the National Renewable Energy Laboratory (NREL) in Golden, CO. Pretreatment of yellow poplar sawdust using the Sunds Hydrolyzer was reported previously *(11)*. Yellow poplar chips (pulp chip size) were pretreated at 0.55% sulfuric acid and 170°C for 15 min using a Sunds Hydrolyzer installed at the Tennessee Valley Authority pilot plant (Muscel Shoals, AL). The water-insoluble fraction of the yellow poplar chips was 71.8% on a dry wt basis, and the soluble solids concentration of the liquid fraction of the pretreated material was 144.6 g/L.

Table 1
Feedstock Composition and Theoretical Component Yields of Pretreated Softwood

Component	Feedstock composition (%)	Theoretical yield after pretreatment (%)
Glucan	43.2	
Unconverted		90.5
To monomeric glucose		10.9
To oligomeric glucose		1.1
To HMF[a]		0.3
Unaccounted for		–2.7
Mannan	11.5	
Unconverted		9.8
To monomeric mannose		74.0
To oligomeric mannose		12.3
Mannan to HMF[a]		2.2
Unaccounted for		+1.7
Galactan	4.3	
Unconverted		29.3
To monomeric galactose		61.3
To oligomeric galactose		10.1
Unaccounted for		–0.7
Xylan	7.7	
Unconverted		15.2
To monomeric xylose		76.4
To oligomeric xylose		8.9
To furfural		5.3
Unaccounted for		–5.9
Arabinan	2.2	
Unconverted		9.4
To monomeric arabinose		96.1
To oligomeric arabinose		9.9
Unaccounted for		–15.3

[a]5-hydroxymethyl-2-furaldehyde.

Effect of Water Temperature on Extraction of Pretreated Softwood

To determine the effect of water temperature on the extraction of soluble solids from the pretreated softwood, stagewise batch extraction was carried out in a glass beaker. For a single-batch extraction, 50 g (wet wt) of pretreated softwood chips (68.3% moisture content) was mixed with 280 mL of deionized water at 25, 40, 60, and 80°C. The slurry was stirred for 2 min, then filtered using a vacuum Buchner filter to separate the liquid from insoluble solids. The extracted solids were dried in a 105°C oven overnight. The soluble solids recovery was determined by subtracting the dry wt of the extracted solids from the dry wt of the starting material. Data were also collected for multiple (as many as five), consecutive batch extrac-

Table 2
Sugar Composition of Liquid Fraction
of Starting Pretreated Softwood and Liquid Extract from Continuous Countercurrent Extraction (g/L)

Liquid	Cellobiose[a]	Glucose	Xylose	Galactose	Arabinose	Mannose
Liquid fraction of pretreated softwood	1.9	17.4	22.6	9.4	7.7	32.4
Extract from L/IS = 2.1	ND	13.3	16.4	7.8	6.2	27.8
Extract from L/IS = 3.4	ND	7.6	10.9	5.2	4.3	17.1
Extract from L/IS = 5.6	ND	5.4	7.3	4.2	2.0	10.2

[a]ND, not detected by HPLC.

tions. For a two-batch extraction, the filtered solids obtained from the first extraction were reslurried with 280 mL of deionized water, stirred, and filtered. This was repeated as many times as the number of extraction batches before the extracted solids were dried in the 105°C oven. The weight ratio of added water to dry insoluble solids (L/IS) was 24 for the single-batch extraction, 47 for the two-batch extraction, and 118 for the five-batch extraction. At 80°C, the resulting soluble solids concentrations were 1.2, 0.25, 0.11, 0.05, and 0.02% (w/w) corresponding to the number of extraction of 1, 2, 3, 4, and 5, respectively.

Drainage Rate of Pretreated Biomass

The continuous countercurrent screw extractor used in the present study relies on percolation of water by gravity through the pretreated biomass. If the pretreated biomass has poor water drainage properties (i.e., very slow drainage rate), channeling or blockage may occur inside the extractor, which can result in low sugar recovery or low throughput. Therefore, bench-scale percolation tests were performed using silicone columns to compare the water drainage rates for three pretreated materials: softwood chips, yellow poplar sawdust, and yellow poplar chips.

Pretreated wood residues (15.6 g on a dry wt basis) were placed in a 2.5-cm (1-in.) diameter × 30.5-cm (12-in.) high silicone column, which was fitted with a filter at the bottom. Hot water at 60°C was added to the top of the column. To keep the total slurry concentration in the column in the first percolation batch (15.2% on a dry wt basis) constant per pretreated material, the weight ratio of added water to total dry solids (L/S) was varied depending on pretreated materials. The L/S ratios in the first batch were 3.4, 4.4, and 3.9 for the pretreated softwood, yellow poplar sawdust, and yellow poplar chips, respectively. Seven consecutive percolations, each with the same amount of water, were performed on the same column to determine whether the drainage rate changed as the amount of water used increased. The time period required for the liquid to completely drain from the column was recorded. The average drainage rate was calculated by dividing the mass of liquid collected by the draining time.

Countercurrent Extraction of Pretreated Biomass

Figure 3 shows a schematic diagram of the pilot-scale continuous countercurrent extractor designed by NREL. A 10-cm (4-in.) diameter × 305-cm (10-ft) long, U-trough screw conveyor, driven by a Link-Belt® drive (Rexnord, Philadelphia, PA), was purchased from FMC (Tupelo, MS) and modified for the purpose of this process. The helical screw has 36 short-pitch flights. Half-inch holes were drilled into the flights and mixing paddles were installed between the flights to improve liquid/solid contact. Because the solids discharge opening was 31 cm (1 ft) from the top of the conveyor, the effective length of the extraction zone was only 274 cm (9 ft). The total working volume of the extractor was 28 L. To minimize channel-

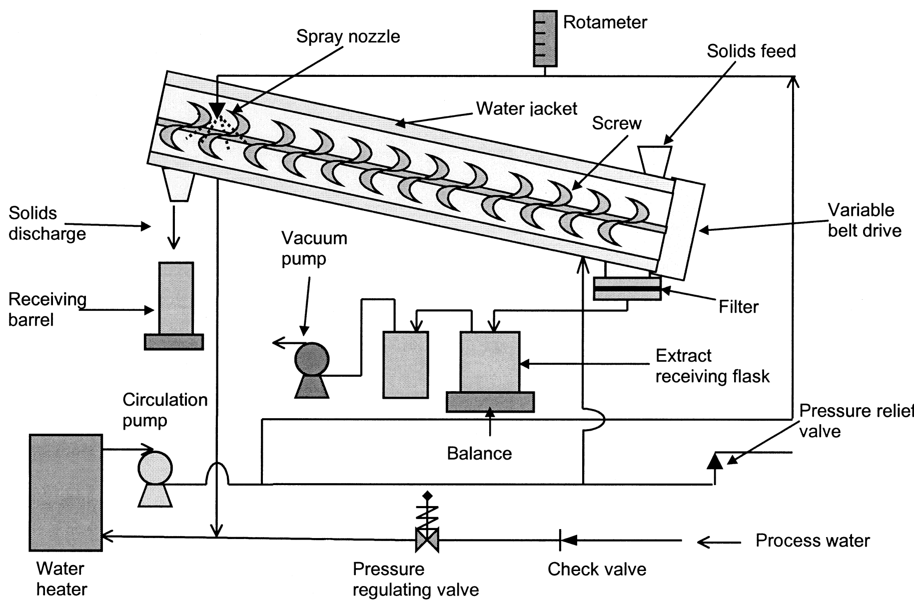

Fig. 3. Schematic of the pilot-scale continuous countercurrent extractor.

ing of water along the bottom of the trough, the screw extractor was mounted with an inclined angle of 50° from horizontal.

At the beginning of a run, a batch of pretreated softwood was loaded into a constant volumetric feeder (Acrison® feeder BDFM; Acrison, Moonachie, NJ). Process water was heated in the water heater to 60°C, then circulated through the screw conveyor jacket. When the return water temperature reached a steady-state value of about 57°C, the feeder was then switched on and set at a predetermined feed rate to begin to introduce pretreated wood into the bottom of the extractor. The conveyor drive was then activated and set at a predetermined forward speed such that the flights were less than 50% filled with pretreated wood. At about the same time, a split stream of hot water was metered through a rotameter and sprayed on top of the pretreated material through a spray nozzle installed approx 31 cm (1 ft) upstream of the solids discharge opening. The extracted liquid passed through coarse filters at the bottom of the extractor and was collected every 5 min into a flask connected to a vacuum pump. The extracted solids were discharged into a barrel via the solids discharge chute at the top of the extractor. The Acrison feeder, extract receiving flask, and barrel for receiving discharged solids were placed on electronic balances, and their weight changes were recorded every 5 min. Each extraction run lasted 110–120 min. Steady state (i.e., no appreciable change in pH of the extracted solids) was obtained after approx 60 min. At the end of each run, samples of solids inside the extractor were taken at 2-ft intervals and analyzed for insoluble solids and percentage of soluble solids extracted.

Analysis of Extracted Solids and Liquid

The concentration of monomeric sugars in the extract was analyzed by high-performance liquid chromatography (HPLC) using the same method for hydrolysate liquor analysis as described previously *(12)*. The fraction of insoluble solids (FIS) of feed materials and extracted solids were determined to find the amount of solubles extracted from pretreated wood by the screw extractor. The FIS is defined as the dry weight ratio of water-insoluble solids over the unwashed solids.

To determine the FIS, approx 7 L of tap water at 40°C was added to solids with known total weight and solid content (approx 180 g of total dry solids or 120 g of insoluble solids for pretreated softwood) in a container. The resulting slurry was mixed vigorously with a portable mixer for 5 min and left standing for 5 min. The approximate L/S and L/IS ratios of the slurry were 44 and 58, respectively. The slurry was filtered with a 24-cm diameter glass-fiber filter (1.5-μm particle retention, Whatman grade 934-AH; Whatman, Maidstone, England) under a vacuum and resuspended with tap water for the next washing. These procedures were repeated at least three more times or until the pH of the slurry was higher than 6.0, and the solids were then washed once more with 40°C deionized water. The total L/S and L/IS ratios used in determining the FIS of pretreated softwood were 220 and 290, respectively. All the washed solids were recovered, mixed, and weighed. Three representative samples were dried overnight in a 105°C oven to determine the solid content of the washed solids. The FIS values for pretreated biomass are generally in the 0.65–0.80 range. We define 100% soluble solids (or solubles) recovery as $100 \times (1 - \text{FIS})$. The percentage of solubles recovery yields of partially extracted solids were calculated according to the following equation:

$$\text{Percentage of solubles recovery} = [(1 - \text{FIS}_i)/(1 - \text{FIS}_0)] \times 100 \qquad (1)$$

in which FIS_i is the FIS of the partially extracted material, and FIS_0 is the FIS of the starting material.

Fitting Empirical Equation to Experimental Data

The following empirical equation was fitted to the experimental data to predict the recovery yield of solubles against the extractor length and the L/IS ratio:

$$R = 100 - a\exp(-bL) \qquad (2)$$

in which R is the recovery yield of solubles (%), a and b are constants, and L is either the length of extractor or L/IS ratio. Nonlinear regression was carried out using the scientific graphing software SigmaPlot® (Jandel, Chicago, IL) to determine constants a and b with different operating parameters.

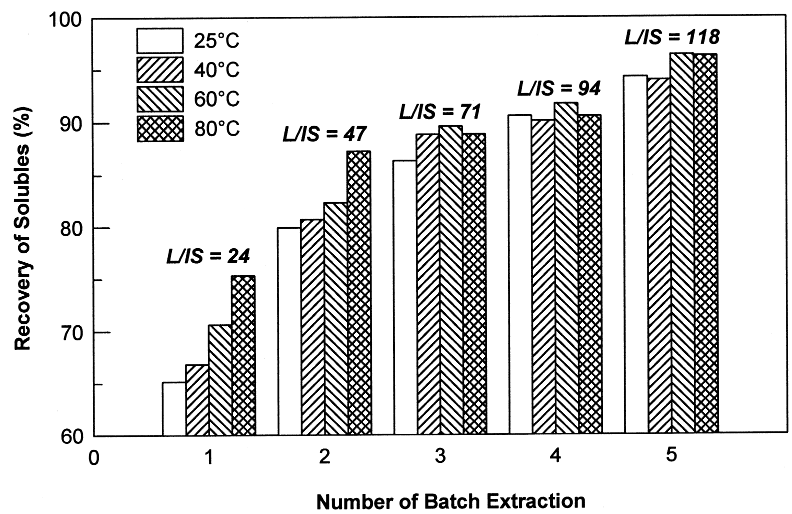

Fig. 4. Effect of wash water temperature on the extraction of solubles from pretreated softwood.

Results and Discussion

Effect of Water Temperature on Extraction of Pretreated Softwood

As shown in Fig. 4, the recovery of solubles from the pretreated softwood forest thinnings increased significantly as the wash water temperature was raised from 25 to 80°C. This effect of water temperature was especially pronounced when low amounts of water were used such as in single- and double-batch extractions, which were carried out with an L/IS ratio of 24 and 47, respectively. Therefore, it is important to use hot water in countercurrent extraction in which a low L/IS ratio is maintained to obtain high extraction efficiency. In the pilot-scale continuous extraction experiments, 57°C water was sprayed on the pretreated biomass and 60°C water was circulated through the screw conveyor jacket.

Comparison of Drainage Rates

Pretreated biomass has different particle sizes depending on the particle size of the starting materials and the pretreatment conditions. If the particle size is too small to give an adequate drainage rate, channeling and blockage of liquid flow may become serious problems for countercurrent screw extractors. In the present study, drainage tests were performed on three different pretreated wood residues; Figure 5 shows the results. The drainage rate through pretreated softwood chips was significantly higher than those obtained with pretreated yellow poplar sawdust and chips, which contain a large amount of fines. This higher drainage rate of the pretreated softwood was most likely attributable to its large particle sizes in comparison to the pretreated yellow poplar materials. The increase in

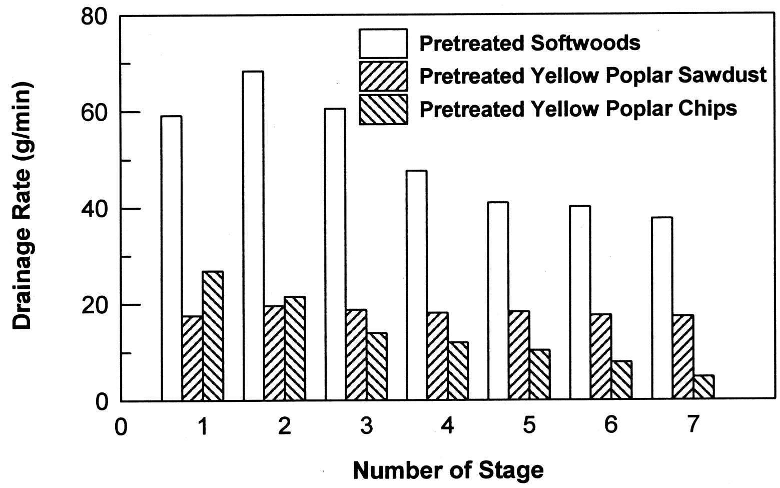

Fig. 5. Comparison of water drainage rates for different pretreated materials using a 1-in. (2.5-cm) diameter × 12-in. (30.5-cm) high column.

drainage resistance probably was caused by the compaction of the bed of washed materials. The bed heights of both the pretreated yellow poplar residues shrank approx 40% after four consecutive extraction batches, whereas that of the pretreated softwood shrank about 25%. A similar packing problem in a column extractor was also reported in oil extraction from fine soybean flour *(13)*. The drainage rates of pretreated wood residues generally decreased as the number of percolation batches increased and later on remained constant. This was probably because water-absorbed wood matrix hampered the water drain in the packed column. Basing our selection on the results of the drainage tests, we chose pretreated softwood for running extraction experiments with the countercurrent extractor. The two pretreated yellow poplar materials with lower water drainage rates will be considered in future studies.

Countercurrent Extraction of Pretreated Softwood Forest Thinnings

Countercurrent extraction of pretreated softwood forest thinnings was performed at three different L/IS ratios and a fixed solid feed rate of approx 220 g/min (228, 209, and 234 g/min for L/IS = 2.1, 3.4, and 5.6, respectively). The L/IS ratio was determined by dividing the amount of water in the extract by the amount of insoluble solids in solids feed. The wash water flow rate was varied for different L/IS ratios (175, 240, and 400 g/min for L/IS = 2.1, 3.4, and 5.6, respectively) and the solids feed rate was kept constant for all runs. Because the screw rotation speed was fixed at 20 rpm for the entire extraction runs, the average solids residence times in the extractor were essentially the same (approx 20 min) for all runs.

Figure 6 shows the concentrations of solubles and pH of extracts recovered during the steady-state operation of the continuous countercurrent extractor at various L/IS ratios. The concentration of solubles in the

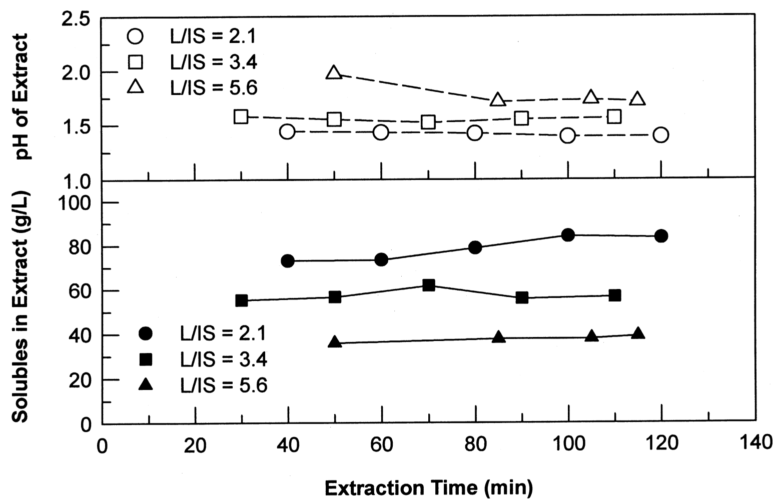

Fig. 6. Extract pH and concentration of solubles in extract recovered from the bottom of the extractor in continuous countercurrent extraction of pretreated softwood at different L/IS ratios.

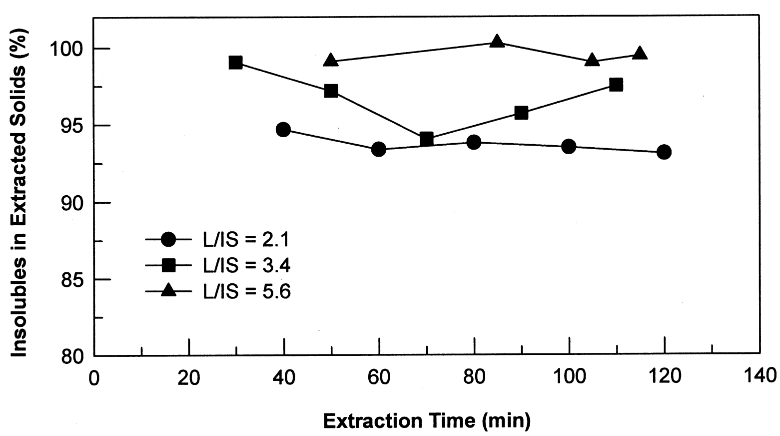

Fig. 7. Percentage of insolubles in extracted solids discharged from the top of the extractor in continuous countercurrent extraction of pretreated softwood at different L/IS ratios.

extract decreased as the L/IS increased because of dilution of the sugar solution contained in the pretreated wood residues at higher water flow rates. Therefore, it is necessary to keep the L/IS ratio low to achieve high solute concentrations in the extract as long as the extraction efficiency is maintained at an adequate level. The increase in extract pH with the L/IS ratio also indicates the dilution effect at higher water flow rates. Table 2 lists the sugar composition of the liquid fraction of the starting pretreated material (i.e., before extraction) and extracts collected from the countercurrent extraction runs at the different L/IS ratios. As expected, the sugar concentration decreased as the L/IS ratio was raised.

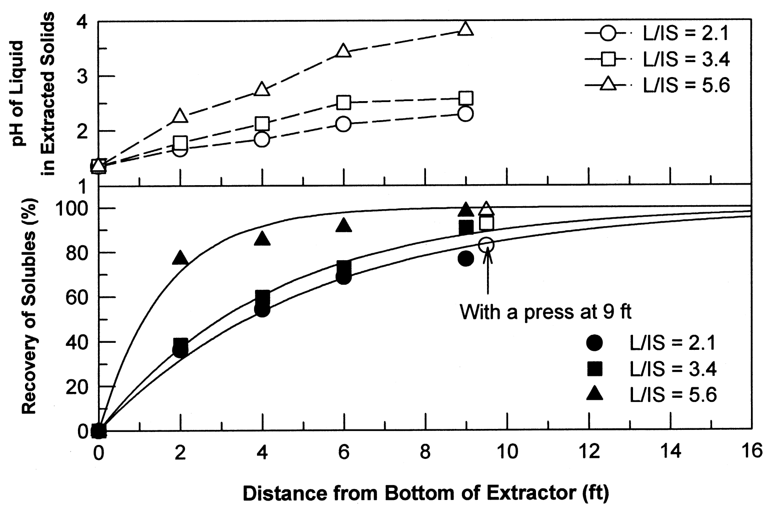

Fig. 8. Solubles recovery and pH of liquid in extracted solids at different locations in the continuous countercurrent extractor operated at steady state after 120 min. Closed symbols indicate experimental data and lines represent the predicted curves.

Figure 7 illustrates the percentage of insolubles in extracted solids discharged during the steady-state operation of the countercurrent extractor. Higher FIS (i.e., insoluble fraction in extracted solids) values were obtained at higher L/IS ratios. This result indicates that higher amounts of soluble solids were extracted at higher L/IS ratios. The lower value of percentage of insolubles in extracted solids at 70-min extraction time from L/IS = 3.4 can be attributed to a disruption in steady-state operation when the clogged filters were replaced. The gradual decline in FIS value for the run of L/IS = 3.4 was most likely caused by plugging of the filters at the bottom of the extractor. After the filter was replaced at 70 min, the extraction efficiency improved, as indicated by the rising FIS trend. We installed a different type of filter for the other two runs and did not observe severe plugging problems.

Operating Line

To establish the operating line of the recovery of solubles with respect to location in the extractor, extracted solids remaining in the extractor were recovered at the end of each extraction run and analyzed for percentage of solubles extracted. The experimental data are indicated by closed symbols in Fig. 8. The solid lines represent the best-fitted operating lines to the experimental data by the empirical equation (Eq. 2).

The percentage of solubles recovery increased as the distance from the bottom of the extractor increased. This was confirmed by an increase in pH of the liquid in the extracted solids. At 274 cm (9 ft) from the bottom of the extractor, where solids were discharged, the solubles recoveries for L/IS ratios of 5.6, 3.4, and 2.1 were 98, 91, and 77%, respectively. In plant opera-

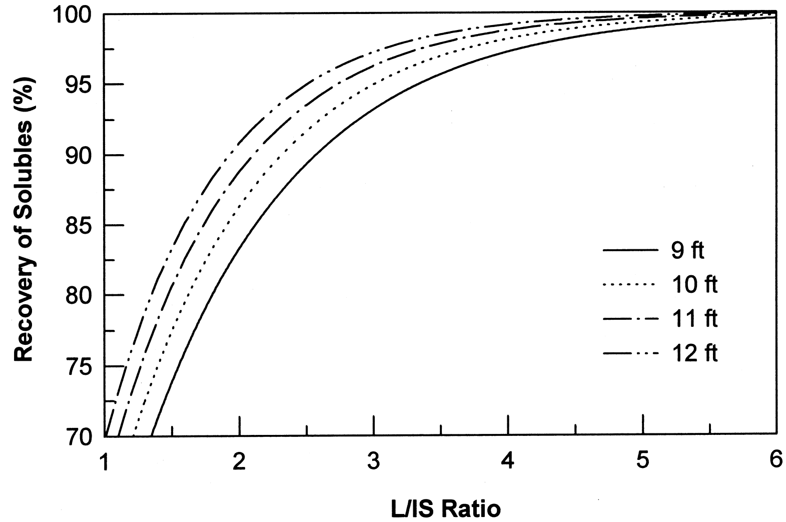

Fig. 9. Prediction of soluble recovery with respect to L/IS ratio for continuous countercurrent extraction of the pretreated softwood with different extractor lengths.

tion, assuming that the extracted solids would be pressed to approx 45% (w/w) solid content to recover the entrained solubles and the obtained liquid returned to the extractor, one would expect a slight enhancement in the solubles recovery, as represented by the open symbols in Fig. 8. The soluble recovery was higher at a higher L/IS ratio for the same extractor length. The values of constants *a* and *b* for the empirical equation used (Eq. 2) for plotting the operating lines in Fig. 8 were estimated to be 99.9120 and 0.1910, 99.9938 and 0.2327, and 99.9710 and 0.6251 for L/IS = 2.1, 3.4, and 5.6, respectively.

Figure 9 presents the prediction of soluble recoveries when the length of the extractor is extended to increase the extraction stages. The estimated values of constants *a* and *b* for Eq. 1 used in plotting the predicted curves in Fig. 9 were 99.9950 and 0.8937, 99.9969 and 0.9926, 99.9981 and 1.0920, and 99.9988 and 1.1916 for extractor lengths of 274.3, 304.8, 335.3, and 365.8 cm, respectively (9, 10, 11, and 12 ft, respectively). A 12-ft extractor with the same configuration as the current extractor used in this work is expected to achieve >95% recovery of solubles at an L/IS ratio of 3.0. This predicted performance is better than the predicted 93.8% soluble recovery for the three-stage stagewise countercurrent washer mentioned earlier (Fig. 2).

Conclusion

We have demonstrated that continuous countercurrent extraction of hemicellulosic sugars from pretreated softwood residues using a pilot-scale screw extractor can be effectively achieved. The soluble recovery yield decreased as L/IS ratio was reduced. The empirical equation predicts that adequate recovery of soluble sugars can be obtained in the low-range L/IS ratio of 2.5–3.0, if the length of the extractor is extended to about 366 cm (12 ft).

Acknowledgment

This work was funded by the U.S. Department of Energy, Office of Fuels Development.

References

1. Nguyen, Q. A., Tucker, M. P., Keller, F. A., and Eddy, F. P. (2000), *Appl. Biochem. Biotechnol.* **84–86,** 561–576.
2. Nguyen, Q. A. and Aden, A. (1999), Report no. 4083, National Renewable Energy Laboratory, Golden, CO.
3. Schell, D. (1997), Report no. 4115, National Renewable Energy Laboratory, Golden, CO.
4. Schwartzberg, H. G. (1980), *Chem. Eng. Prog.* **76,** 67–85.
5. Smook, G. A. (1992), *Handbook for Pulp and Paper Technologies*, 2nd ed., Angus Wilde, Vancouver, BC.
6. Rundle, K. W. (1989), US patent 4,873,095.
7. Noah, K. S. and Linden, J. C. (1989), *Trans. ASAE* **32,** 1419–1425.
8. Nguyen, Q. A. (1993), Canadian patent 1,322,366.
9. Brinkley, C. R. and Wiley, R. C. (1978), *J. Food Sci.* **43,** 1019–1023.
10. Gunasekaran, S., Fisher, R. J., and Casmir, D. J. (1989), *J. Food Sci.* **54,** 1261–1265.
11. Tucker, M. P., Farmer, J. D., Keller, F. A., Schell, D. J., and Nguyen, Q. A. (1998), *Appl. Biochem. Biotechnol.* **70–72,** 25–35.
12. Nguyen, Q. A., Tucker, M. P., Boynton, B. L., Keller, F. A., and Schell, D. J. (1998), *Appl. Biochem. Biotechnol.* **70–72,** 77–87.
13. Nieh, C. D. and Snyder, H. E. (1991), *J. Am. Oil Chem. Soc.* **68,** 246–249.

Permission to Use Copyrighted Material

☒ Yes I give the National Renewable Energy Laboratory (NREL) of Golden, Colorado, irrevocable permission to post the following onto its site on the World Wide Web

☐ No

Name of Document, Report, Article, etc.:

Continuous Countercurrent Extraction of Hemicellulose from Pretreated Wood Residues

☒ Yes In addition to posting on the Web, I give NREL permission to distribute hard copies of the document through its National Alternative Fuels Hotline in response to requests.

☐ No

When posting on the Web, NREL will credit the copyright holder thus: "Posted on this site with permission from ." When distributing hard copies, NREL will label the document "Reprinted with permission from .

I deny permission to post the document on the Web or to distribute hard copies, but give permission for the document to be abstracted and cited in NREL's database, along with information about obtaining the document directly from the copyright holder. The document can be obtained by writing:

I represent and warrant that I have the full right and authority to grant the rights herein granted and that no one else's permission is required.

Printed name Thomas B. Lanigan

Thomas B Lanigan

Signed name

Title and company VP, Mktg + Sales Humana Press

6/27/01

Date

Created February 1997